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CN1198453 (reference document 2)

Claims

1. A modified poly ether-ether-ketone composite material, which comprises poly ether-ether-ketone and flock, characterized in that said composite material still comprises a thermotropic liquid crystal polymer, the components and their contents (part by weight) being as follows:

Component	Content
Poly ether-ether-ketone	40-80
Thermotropic liquid crystal polymer	5-30
Flock	10-40

2. The poly ether-ether-ketone composite material according to claim 1, characterized in that the content of said poly ether-ether-ketone is 55-70 parts by weight.

3. The poly ether-ether-ketone composite material according to claim 1, characterized in that the content of said flock is 15-30 parts by weight.

4. The poly ether-ether-ketone composite material according to claim 1, characterized in that said thermotropic liquid crystal polymer is a backbone-type thermotropic liquid crystal polymer.

5. The poly ether-ether-ketone composite material according to claim 4, characterized in that said backbone-type thermotropic liquid crystal polymer is a backbone-type aromatic copolyester.

6. The poly ether-ether-ketone composite material according to claim 1, characterized in that said flock is a short glass fiber or short carbon fiber.

7. Any poly ether-ether-ketone composite material according to claim 5, characterized in that said backbone-type aromatic copolyester is the random copolyester of para-hydroxybenzoic acid and 6-hydroxy-2-naphthoic acid.

8. Any poly ether-ether-ketone composite material according to claim 5, characterized in that said backbone-type aromatic copolyester is the random copolyester of para-hydroxybenzoic acid and diethyl terephthalate.

9. The short glass fiber according to claim 6, characterized in that it is a

glass fiber, the surface of which is treated with a coupling agent or graft copolymer.

10. The short carbon fiber according to claim 6, characterized in that it is a carbon fiber, the surface of which is treated by oxidation or electrodeposition.

A poly ether-ether-ketone composite material modified with thermostatic liquid crystal polymers

The present invention relates to a thermoplastic material reinforced with flock, particularly to a poly ether-ether-ketone composite material modified with thermostatic liquid crystal polymer and reinforced with flock.

Poly ether-ether-ketone (PEEK) is a special engineering plastic with excellent comprehensive properties, for example, it possesses thermotolerance and chemical stability of thermosetting plastics and moldability of thermoplastic plastics at the same time. Compared to the unreinforced poly ether-ether-ketone, the poly ether-ether-ketone reinforced with flock exhibits higher strength, modulus, and thermotolerance. Polymer Composites Vol. 17, P468, 1996 reported a poly ether-ether-ketone composite material reinforced with short glass fiber and short carbon fiber, the mechanical property of which was apparently superior to the poly ether-ether-ketone substrate. The melting point of poly ether-ether-ketone and the viscosity of its molten mass are rather high, and the dependence of its melt fluidity on temperature is not strong, so it requires higher molding temperatures (the temperature of the charging barrel is controlled at about 350-400°C) and injecting pressures in processing. Processing of the poly ether-ether-ketone composite material reinforced with flock is difficult because the melt fluidity becomes poorer due to the presence of flock in the system. What is more unfavorable is that the high content of reinforcing fiber enhances the friction between fiber and fiber, fiber and the equipment, causing severe rupture of the reinforcing fiber and reduction of the reinforcing effect.

The objective of the present invention is to overcome the shortcomings of poor melt fluidity, wear to the equipment, and significant rupture of flock present in the poly ether-ether-ketone system reinforced with flock, and provide a poly ether-ether-ketone composite material modified with thermostatic liquid crystal polymer and reinforced with flock, this material having good processing and mechanical properties.

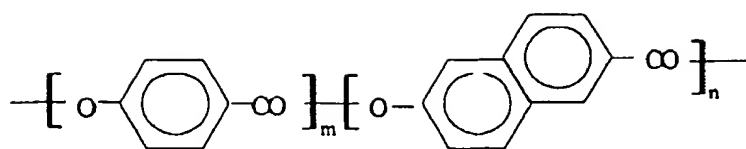
The feedstocks of the poly ether-ether-ketone composite material of the present invention are poly ether-ether-ketone, flock, and thermotropic liquid crystal polymer, and the components and their contents are as follows:

Component	Content (parts by weight)
Poly ether-ether-ketone	40-80 (most preferably 55-70)
Thermotropic liquid crystal polymer	5-30
Flock	10-40 (most preferably 15-30)

The aforesaid flock is a surface treated carbon fiber (CF) or glass fiber (GF). The surface of the glass fiber may be treated with a coupling agent or graft polymer. Alternate copolymer of vinyl acetate-maleic anhydride or alternate copolymer of styrene-maleic anhydride may be deposited on the surface of carbon fiber by surface oxidation or electrodeposition. The aforesaid thermostatic liquid crystal polymer (TCLP) is backbone-type aromatic copolyester with a melting range between 190°C and 350°C, exhibiting an interlaced structure under a micropolariscope. For example, the random copolyester of para-hydroxybenzoic acid with glycol phthalate has melting points ranging from 250°C to 350°C and the following structural formula:



TLCP may also be random full-aromatic copolyester of para-hydroxybenzoic acid with 6-hydroxy-2-naphthoic acid with a melting point ranging from 270°C to 350°C and the following structural formula:



The preparation process of the poly ester-ester-ketone composite material of the present invention proceeds according to the following steps:

10-20 parts of thermostatic liquid crystal polymer (TLCP), 50-80 parts of poly ester-ester-ketone substrate, and 10-40 parts of flock are mixed, extruded, and pelleted through a single- or double-screw extruder at 370°C-400°C, and then subjected to injection-molding.

In the melt processing of the poly ester-ester-ketone composite material of the present invention, which is modified with thermostatic liquid crystal polymer and reinforced with carbon fiber, both TLCP and poly ester-ester-ketone are molten mass. The rather strong shear thinning rheological behavior of TLCP lowers the viscosity of the molten mass, reduces the torque of the screw rod in processing (see Table I. The rheological behavior of the molten mass was characterized with HAAKE RC 90 torque rheometer, showing that the balance torque is directly proportional to the viscosity and the total torque at a certain moment is directly proportional to the work consumed so far for mixing), reduces the wear of the extruder and injection-molding machine by the material, and meanwhile, reduces the rupture rate of the flock in processing, raises the average length/diameter ratio of the flock in the composite material (see Table II), and enhances the mechanical properties of the poly ester-ester-ketone composite material (see Table III).

Table I shows the balance torque $M_{360^\circ\text{C}}$ and total torque at the 5th minute of the composite materials with different weight ratios of thermostatic liquid crystal polymer to carbon fiber to poly ester-ester-ketone when they were mixed in the HAAKE RC 90 torque rheometer (the rotation rate of the rotor was 40 rpm).

Table II shows the number average length and number average length/diameter ratio of the carbon fiber in the injection-molding product of

the composite materials with different weight ratios of thermostatic liquid crystal polymer to carbon fiber to poly ester-ester-ketone.

Table III shows the tensile mechanical properties of the composite materials with different weight ratios of thermostatic liquid crystal polymer to carbon fiber to poly ester-ester-ketone.

Table I

Sample	Balance torque $M_{360^{\circ}\text{C}}$ (Nm)	Total torque at 5 th minute (Nm-min)
PEEK	5.8	31
PEEK/CF 85/15	9.8	49
PEEK/CF/TLCP 70/15/15	8.8	44
PEEK/CF 70/30	13.8	68
PEEK/CF/TLCP 55/30/15	9.5	51

It can be seen from Table I that addition of TLCP improved the processing properties of the system relative to the carbon fiber/poly ether-ether-ketone system.

Table II

Sample	Number average length of carbon fiber (μm)	Number average length to diameter ratio of carbon fiber
PEEK/CF 85/15	123.6	16.5
PEEK/CF/TLCP 70/15/15	164.8	22.0
PEEK/CF 70/30	103.5	13.8
PEEK/CF/TLCP 55/30/15	122.9	16.4

It can be seen from Table II that addition of TLCP reduced the rupture rate of the carbon fiber and raises the number average length/diameter ratio of carbon fiber in extruding and injection-molding relative to the carbon fiber/poly ether-ether-ketone system.

Table III

Sample	Tensile strength (MPa)	Tensile modulus (GPa)
PEEK	91	2.9
PEEK/CF 85/15	141	5.2
PEEK/CF/TLCP 70/15/15	154	5.6
PEEK/CF 70/30	*	*
PEEK/CF/TLCP 55/30/15	183	8.8

Note: the PEEK/CF 70/30 system has too high a viscosity to be molded on the CS-183 injection-molding machine

It can be seen from Table III that addition of TLCP improved the mechanical properties of the system relative to the carbon fiber/poly ether-ether-ketone system.

Example 1

15 parts of PHB/HNB (73/27) thermostatic liquid crystal polymer, 15 parts of surface treated carbon fiber, and 70 parts of poly ether-ether-ketone (the limit viscosity determined in concentrated sulfuric acid solution was $[\eta]=0.83$ dl/g, $T_g=143^\circ\text{C}$, $T_m=334^\circ\text{C}$) were molten and mixed on the CS-194A extruder at a rotor temperature of 380°C and a die orifice temperature of 370°C . The extruded bars were pelleted after cooling and the resultant pellets were injected into a tensile strip specimen of ASTM standard with a rectangular cross section and a dumbbell shape on the CS-183 MINI MAX injection-molding machine under a molten mass temperature of 380°C and a

modulus temperature of 150°C. The balance torque $M_{360^{\circ}\text{C}}$ was 8.8 Nm and the total torque at the 5th minute was 44 Nm-min when the above components were mixed in the HAAKE RC 90 torque rheometer. The tensile strength of the above tensile strip specimen was 154 MPa, the tensile modulus was 5.6 GPa, and the derived number average length/diameter ratio of the carbon fiber in the injected tensile strip specimen was 22.0.

Example 2

15 parts of PHB/HNB (73/27) thermostatic liquid crystal polymer, 30 parts of surface treated carbon fiber, and 55 parts of poly ether-ether-ketone (the limit viscosity determined in concentrated sulfuric acid solution was $[\eta]=0.83$ dl/g, $T_g=143^{\circ}\text{C}$, $T_m=334^{\circ}\text{C}$) were used and the other conditions were the same as Example 1. The balance torque $M_{360^{\circ}\text{C}}$ was 9.5 Nm and the total torque at the 5th minute was 51 Nm-min when the above components were mixed in the HAAKE RC 90 torque rheometer. The tensile strength of the above tensile strip specimen was 183 MPa, the tensile modulus was 8.8 GPa, and the derived number average length/diameter ratio of the carbon fiber in the injected tensile strip specimen was 16.4.

Example. 3

20 parts of PHB/HNB (73/27) thermostatic liquid crystal polymer, 40 parts of surface treated carbon fiber, and 40 parts of poly ether-ether-ketone (the limit viscosity determined in concentrated sulfuric acid solution was $[\eta]=0.83$ dl/g, $T_g=143^{\circ}\text{C}$, $T_m=334^{\circ}\text{C}$) were molten and mixed on the CS-194A extruder at a rotor temperature of 380°C and a die orifice temperature of 370°C. The extruded bars were pelleted after cooling to yield a poly ether-ether-ketone composite material modified with thermostatic liquid crystal polymer and reinforced with short carbon fiber.

Example 4

10 parts of PHB/HNB (73/27) thermostatic liquid crystal polymer, 10 parts of surface treated carbon fiber, and 80 parts of poly ether-ether-ketone (the limit viscosity determined in concentrated sulfuric acid solution was $[\eta]=0.83$ dl/g, $T_g=143^{\circ}\text{C}$, $T_m=334^{\circ}\text{C}$) were molten and mixed on the CS-194A extruder at a rotor temperature of 380°C and a die orifice temperature

of 370°C. The extruded bars were pelleted after cooling to yield a poly ether-ether-ketone composite material modified with thermostatic liquid crystal polymer and reinforced with short carbon fiber.

Comparative Example 1

15 parts of carbon fiber was used to reinforce 85 parts of poly ether-ether-ketone and the other conditions were the same as Example 1. The balance torque $M_{360^{\circ}\text{C}}$ was 9.8 Nm and the total torque at the 5th minute was 49 Nm-min when the above components were mixed in HAAKE RC 90 torque rheometer. The tensile strength of the above tensile strip specimen was 141 MPa, the tensile modulus was 5.2 GPa, and the derived number average length/diameter ratio of the carbon fiber in the injected tensile strip specimen was 16.5.

Comparative Example 2

30 parts of carbon fiber was used to reinforce 70 parts of poly ether-ether-ketone and the other conditions were the same as Example 1. The balance torque $M_{360^{\circ}\text{C}}$ was 13.8 Nm and the total torque at the 5th minute was 68 Nm-min when the above components were mixed in HAAKE RC 90 torque rheometer. The viscosity of the system was too high to be molded in CS-183 MINI MAX injection-molding machine. The derived number average length/diameter ratio of the carbon fiber in the extruded bars was 13.8.

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权利要求书 1 页 说明书 5 页 附图页数 0 页

[54]发明名称 一种用热致液晶聚合物改性的聚醚醚酮
复合材料

[57]摘要

本发明一种用热致液晶聚合物改性的短纤维增强聚醚醚酮复合材料,其中热致液晶聚合物(TLCP)为主链型对羟基苯甲酸和6-羟基-2-萘甲酸的无规全芳香共聚酯,或者为主链型对羟基苯甲酸和对苯二甲酸乙二酯的无规共聚酯,其中短纤维为经表面处理过的碳纤维或玻璃纤维。TLCP的加入,既改善了复合材料的加工性能,又提高了复合材料的力学性能。

权 利 要 求 书

1、一种改性的聚醚醚酮复合材料包括聚醚醚酮、短纤维，其特征在于所述的复合材料还包括热致液晶聚合物，其组分和含量(重量份)如下：

组 分	含量
聚醚醚酮	40 - 80
热致液晶聚合物	5 - 30
短纤维	10 - 40

2、根据权利要求 1 所述的一种聚醚醚酮复合材料，其特征在于所述的聚醚醚酮含量按重量份为 55- 70。

3、根据权利要求 1 所述的一种聚醚醚酮复合材料，其特征在于所述的短纤维的含量按重量份为 15- 30。

4、根据权利要求 1 所述的一种聚醚醚酮复合材料，其特征在于所述的热致液晶聚合物为主链型热致液晶聚合物。

5、根据权利要求 4 所述的一种聚醚醚酮复合材料，其特征在于所述的主链型热致液晶聚合物为主链型芳香共聚酯。

6、根据权利要求 1 所述的一种聚醚醚酮复合材料，其特征在于所述的短纤维为短玻璃纤维或短碳纤维。

7、根据权利要求 5 中任意一种聚醚醚酮复合材料，其特征在于所述的主链型芳香共聚酯为对羟基苯甲酸和 6-羟基-2-萘甲酸的无规共聚酯。

8、根据权利要求 5 中任意一种聚醚醚酮复合材料，其特征在于所述的主链型芳香共聚酯为对羟基苯甲酸和对苯二甲酸乙二酯的无规共聚酯。

9、根据权利要求 6 要求所述的短玻璃纤维，其特征在于它是表面用偶联剂或接枝共聚物进行处理过的玻璃纤维。

10、根据权利要求 6 所述的短碳纤维，其特征在于它是表面用氧化法，或用电沉积方法处理过的碳纤维。

说明书

一种用热致液晶聚合物改性的聚醚醚酮复合材料

本发明涉及一种用短纤维增强的热塑性材料，特别涉及到一种用热致液晶聚合物改性的短纤维增强聚醚醚酮复合材料。

聚醚醚酮 (PEEK) 是一种综合性能优异的特种工程塑料，它兼具有热固性塑料的耐热性、化学稳定性和热塑性塑料的成型加工性。用短纤维增强的聚醚醚酮，与未增强的相比，显示出更高的强度、模量和耐热性。1996 年 Polymer Composites Vol. 17 P468 报道了短玻璃纤维和短碳纤维增强的聚醚醚酮复合材料，该复合材料的力学性能明显优于聚醚醚酮基体。由于聚醚醚酮的熔点较高，熔体粘度也较大，而且其熔融流动性对温度的依赖性不大，所以聚醚醚酮在加工时需要较高的成型温度 (料筒温度需要控制在 350~400°C 左右) 和注射压力。对于短纤维增强聚醚醚酮复合材料来说，由于体系中含有短纤维因而熔融流动性变得更差，使得加工困难。更不利的是，高含量的增强纤维使得纤维与纤维之间、纤维与设备之间的摩擦增加，使得增强纤维发生剧烈折断，降低了增强纤维的增强效果。

本发明的目的是克服短纤维增强的聚醚醚酮体系熔融流动性差、对设备的磨损、短纤维大量折断，而提供一种用热致液晶聚合物改性的短纤维增强聚醚醚酮复合材料，该材料加工性能好而且力学性能高。

本发明的聚醚醚酮复合材料的原料为聚醚醚酮、短纤维和热致液晶聚合物，各原料的组成和含量如下：

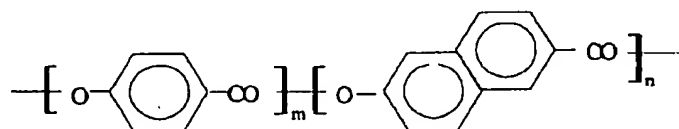
组 分	含量 (重量份)
聚醚醚酮	40 - 80 (最佳 55-70)
热致液晶聚合物	5 - 30
短纤维	10 - 40 (最佳 15-30)

上述的短纤维为经表面处理过的碳纤维 (CF) 或玻璃纤维 (GF)。玻璃纤维表面可以用偶联剂、接枝高聚物进行处理。碳纤维可以用表面氧化法，或电沉积的方法在纤维表面沉积上乙酸乙烯酯-马来酸酐交替共聚物或苯乙烯-马来酸酐交替共聚物。上述的热致液晶聚合物 (TLCP) 为主链型芳香共聚酯，熔融范围为 190°C~

350°C, 在偏光显微镜下呈织态结构。例如对羟基苯甲酸和对苯二甲酸乙二酯的无规共聚酯, 熔点范围为 250°C~350°C, 结构式如下:



TLCP 还可以是对羟基苯甲酸 和 6-羟基-2-萘甲酸 (HNB) 的无规全芳香共聚酯, 熔点范围为 270 °C~350°C, 结构式如下:



本发明的聚醚醚酮复合材料的制法按以下步骤进行:

将热致液晶聚合物 (TLCP) 10~20 份, 与基体聚醚醚酮 50~80 份, 以及短纤维 10~40份, 通过单螺杆或双螺杆挤出机在 370°C~400°C 内共混挤出造粒, 然后注塑成型。

本发明的用热致液晶聚合物改性的碳纤维增强聚醚醚酮复合材料在熔融加工时, TLCP 与聚醚醚酮均为熔体, 由于 TLCP 具有较强的剪切变稀的流变性能, 从而降低了熔体的粘度, 降低了加工时螺杆的转矩 (见表 I, 用 HAAKE RC 90 转矩流变仪表征熔体的流变性能, 其中平衡转矩正比于粘度, 某时刻的总转矩正比于直至该时刻共混所消耗的功), 减少了物料对挤出机和注塑机的磨损, 同时降低了短纤维在加工时的折断率, 提高了短纤维在复合材料中的平均长径比 (见表 II), 提高了聚醚醚酮复合材料的力学性能 (见表 III)。

表 I 为不同重量比的热致液晶聚合物、碳纤维和聚醚醚酮复合材料在 HAAKE RC 90 转矩流变仪中共混时的平衡转矩 $M_{360^\circ\text{C}}$ 和 5 分钟时的总转矩 (转子转速为 40 转/分)。

表 II 为不同重量比的热致液晶聚合物、碳纤维和聚醚醚酮复合材料在注塑制品中碳纤维的数均长度和数均长径比。

表 III 为不同重量比的热致液晶聚合物、碳纤维和聚醚醚酮复合材料的拉伸力学性能。

表 I

样品	平衡转矩 $M_{360^{\circ}\text{C}}$ (Nm)	5 分钟时的总转矩 (Nm·min)
PEEK	5.8	31
PEEK/CF 85/15	9.8	49
PEEK/CF/TLCP 70/15/15	8.8	44
PEEK/CF 70/30	13.8	68
PEEK/CF/TLCP 55/30/15	9.5	51

从表 I 可以看出, 相对于碳纤维/聚醚醚酮体系, TLCP 的加入, 改善了体系的加工性能.

表 II

样品	碳纤维的数均长度 (μm)	碳纤维的数均长径比
PEEK/CF 85/15	123.6	16.5
PEEK/CF/TLCP 70/15/15	164.8	22.0
PEEK/CF 70/30	103.5	13.8
PEEK/CF/TLCP 55/30/15	122.9	16.4

从表 II 可以看出, 相对于碳纤维/聚醚醚酮体系, TLCP 的加入, 降低了碳纤维在挤出和注塑加工中的折断率, 提高了碳纤维的数均长径比.

表 III

样品	拉伸强度 (MPa)	拉伸模量 (GPa)
PEEK	91	2.9
PEEK/CF 85/15	141	5.2
PEEK/CF/TLCP 70/15/15	154	5.6
PEEK/CF 70/30	*	*
PEEK/CF/TLCP 55/30/15	183	8.8

注: PEEK/CF 70/30 体系由于粘度太大而无法在 CS-183 注塑机上成型。

从表 III 可以看出, 相对于碳纤维/聚醚醚酮体系, TLCP 的加入, 提高了体系的力学性能。

实施例 1:

用 15 份 PHB/HNB (73/27) 热致液晶聚合物, 15 份经表面处理的碳纤维与 70 份聚醚醚酮 (在浓硫酸溶液中测得的极限粘度为 $[\eta]=0.83 \text{ dl/g}$, $T_g=143^\circ\text{C}$, $T_m=334^\circ\text{C}$) 在 CS-194A 挤出机上熔融共混, 转子温度为 380°C , 模口温度为 370°C 。挤出条冷却后造粒, 所得粒料在 CS-183 MINI MAX 注塑机上注射出 ASTM 标准的矩形截面的哑铃型拉伸小样条, 熔体温度为 380°C , 模温为 150°C 。上述配方在 HAAKE RC 90 转矩流变仪中共混时的平衡转矩 $M_{360^\circ\text{C}}$ 为 8.8 Nm , 5 分钟时的总转矩为 $44 \text{ Nm}\cdot\text{min}$ 。上述拉伸样条的拉伸强度为 154 MPa , 拉伸模量为 5.6 GPa 。所得注射拉伸样条中碳纤维的数均长径比为 22.0。

实施例 2:

用 15 份 PHB/HNB (73/27) 热致液晶聚合物, 30 份经表面处理的碳纤维与 55 份聚醚醚酮 (在浓硫酸溶液中测得的极限粘度为 $[\eta]=0.83 \text{ dl/g}$, $T_g=143^\circ\text{C}$, $T_m=334^\circ\text{C}$), 其它条件同实施例 1。上述配方在 HAAKE RC 90 转矩流变仪中共混时的平衡转矩 $M_{360^\circ\text{C}}$ 为 9.5 Nm , 5 分钟时的总转矩为 $51 \text{ Nm}\cdot\text{min}$ 。上述拉伸样条的拉伸强度

为 183 MPa, 拉伸模量为 8.8 GPa. 所得注射拉伸样条中碳纤维的数均长径比为 16.4.

实施例3:

用 20 份 PHB/HNB (73/27) 热致液晶聚合物, 40 份经表面处理的碳纤维与 40 份聚醚醚酮 (在浓硫酸溶液中测得的极限粘度为 $[\eta]=0.83$ dl/g, $T_g=143^{\circ}\text{C}$, $T_m=334^{\circ}\text{C}$) 在 CS-194A 挤出机上熔融共混, 转子温度为 380°C , 模口温度为 370°C . 挤出条冷却后造粒, 得到用热致液晶聚合物改性的短碳纤维增强的聚醚醚酮复合材料.

实施例4:

用 10 份 PHB/HNB (73/27) 热致液晶聚合物, 10 份经表面处理的碳纤维与 80 份聚醚醚酮 (在浓硫酸溶液中测得的极限粘度为 $[\eta]=0.83$ dl/g, $T_g=143^{\circ}\text{C}$, $T_m=334^{\circ}\text{C}$) 在 CS-194A 挤出机上熔融共混, 转子温度为 380°C , 模口温度为 370°C . 挤出条冷却后造粒, 得到用热致液晶聚合物改性的短碳纤维增强的聚醚醚酮复合材料.

对比例 1:

用 15 份碳纤维增强 85 份聚醚醚酮, 其它条件同实施例 1. 上述配方在 HAAKE RC 90 转矩流变仪中共混时的平衡转矩 $M_{360^{\circ}\text{C}}$ 为 9.8 Nm, 5 分钟时的总转矩为 49 Nm-min. 上述拉伸样条的拉伸强度为 141 MPa, 拉伸模量为 5.2 GPa. 所得注射拉伸样条中碳纤维的数均长径比为 16.5.

对比例 2:

用 30 份碳纤维增强 70 份聚醚醚酮, 其它条件同实施例 1. 上述配方在 HAAKE RC 90 转矩流变仪中共混时的平衡转矩 $M_{360^{\circ}\text{C}}$ 为 13.8 Nm, 5 分钟时的总转矩为 68 Nm-min. 由于该体系的粘度太大, 以至于无法在 CS-183 MINI MAX 注塑机在注射成型. 所得挤出条中碳纤维的数均长径比为 13.8.